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The effect of zirconia sintering temperature on flexural strength, grain size, and contrast ratio

Stawarczyk, B ; Özcan, M ; Hallmann, L ; Ender, A ; Mehl, A ; Hämmerle, C H F

Abstract: OBJECTIVE: This study investigated the effect of sintering temperatures on flexural strength, contrast ratio, and grain size of zirconia. MATERIALS AND METHODS: Zirconia specimens (Ceramill ZI, Amann Girrbach) were prepared in partially sintered state. Subsequently, the specimens were randomly divided into nine groups and sintered with different final sintering temperatures: 1,300°C, 1,350°C, 1,400°C, 1,450°C, 1,500°C, 1,550°C, 1,600°C, 1,650°C, or 1,700°C with 120 min holding time. Three-point flexural strength (N = 198; n = 22 per group) was measured according to ISO 6872: 2008. The contrast ratio (N = 90; n = 10 per group) was measured according to ISO 2471: 2008. Grain sizes and microstructure of different groups were investigated (N = 9, n = 1 per group) with scanning electron microscope. Data were analyzed using one-way ANOVA with Scheffé test and Weibull statistics ($p < 0.05$). Pearson correlation coefficient was calculated between either flexural strength or contrast ratio and sintering temperatures. RESULTS: The highest flexural strength was observed in groups sintered between 1,400°C and 1,550°C. The highest Weibull moduli were obtained for zirconia sintered at 1,400°C and the lowest at 1,700°C. The contrast ratio and the grain size were higher with the higher sintering temperature. The microstructure of the specimens sintered above 1,650°C exhibited defects. Sintering temperatures showed a significant negative correlation with both the flexural strength ($r = -0.313$, $p < 0.001$) and the contrast ratio values ($r = -0.96$, $p < 0.001$). CONCLUSIONS: The results of this study showed that the increase in sintering temperature increased the contrast ratio, but led to a negative impact on the flexural strength. CLINICAL RELEVANCE: Considering the flexural strength values and Weibull moduli, the sintering temperature for the zirconia tested in this study should not exceed 1,550°C.

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Effect of sintering temperature on optical and mechanical properties of Y-TZP ceramic

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Abstract

Objective: This study determined the effect of sintering temperature on flexural strength, relative translucency, and grain size of a Y-TZP ceramic. *Materials and Methods:* For all three used test methods, the specimens were prepared in white state (Ceramill ZI, Amann Girrbach, Koblach, Austria). The specimens were then divided into nine groups. Specimens in each group were sintered at a heat rate of 8°C/min. The final sintering temperature ranged between 1300°C and 1700°C with intervals of 50°C and a holding time of 120 min for each group. Three-point flexural strength (N=198; n=22 per group) was measured according to ISO 6872: 2008. The relative translucency (N=90; n=10 per group) was measured using a spectrophotometer according to ISO 2471: 2008. Grain sizes of the differently sintered zirconia were investigated (N=9, n=1 per group) using a Scanning Electron Microscopy. All data were analysed with one-way ANOVA followed by a post-hoc Scheffé test ($p < 0.05$) and the simple linear correlation was evaluated. *Results:* The highest flexural strength was observed from specimens sintered between 1400°C and 1550°C. The translucency and the grain size increased with the sintering temperature. The microstructure of specimens sintered above 1650°C showed critical defects. *Conclusions:* The results of this study clearly showed that the increase in sintering temperature increase the translucency, but showed a negative impact on the mechanical properties.

Keywords: zirconia, ceramic, flexural strength, translucency, grain size, sintering parameters, sintering temperature

1. Introduction

Zirconia exhibits polymorphic properties meaning that same chemical composition exists in different atomic arrangement [1,2]. Among the crystallographic structures, the monoclinic phase is stable up to 1170°C. It transforms into the tetragonal phase, which is stable up to 2370°C. The cubic phase on the other hand, exists up to the melting point of 2680°C [1,2]. The tetragonal form for metastable zirconia in biomedical applications could be achieved at room temperature by alloying zirconia with other oxides (stabilizers), e.g. CaO [3], MgO [4], Y₂O₃ [5-8] and CeO₂ [9]. Y₂O₃ (approximately 3 mol%) is the most widely used stabilizer for dental zirconia (Y-TZP) [2]. In response to mechanical stimuli such as tensile stress at the crack tips, the stabilized tetragonal zirconia transforms to the more stable monoclinic phase with a local increase in volume of approximately 4-5 vol% [10]. This increase in volume closes the crack tips, effectively blunting crack propagation [10]. It is this transformation-toughening process, which gives zirconia its strength and toughness, exceeding all currently available dental ceramics [10]. From an optical point of view, Y-TZP is white and can be easily coloured by addition of traces of rare earth elements [2]. Zirconia, with its high degree of crystalline content, has a maximum light scattering effect. Thus, they appear relatively opaque under visible light [11].

Due to difficult handling of the material zirconia, the developments in CAD/CAM technology has made it possible use zirconia in dentistry. This technology enables complex forms to be milled out of zirconia blanks. Two types of zirconia milling processes are currently available: soft-milling (“white state”) and hard-milling (HIP state). Since soft-milling is relatively easily and quick, most manufacturers chose this method for the fabrication of fixed dental prosthesis (FDPs). Soft-milling involves machining enlarged frameworks out of pre-sintered blanks of zirconia, also

called the “white” state. These are then sintered to their full strength, which is accompanied by shrinkage of the milled framework by approximately 25% to the desired dimensions. Between manufacturers, the sintering parameters of zirconia show variations. After sintering, zirconia frameworks can be veneered using either layering or overpressed technique.

Zirconia FDPs are feasible for molar region, due to their high flexural strength and fracture toughness [12-15]. Limited number of clinical studies reported seldom zirconia framework fractures but more often chipping of the veneering ceramic [16-20]. In fact, the stability of the complete system consisting both the zirconia framework and the veneering ceramic is of clinical importance.

In order to decrease the costs and at the same time overcome the chipping problem, it is possible today to produce zirconia FDPs without veneering ceramic. Such monolithic zirconia FDPs require higher translucency with superior mechanical properties especially when they are to be used in the anterior region. Framework translucency is therefore one of the primary factors with respect to aesthetic properties in the selection of dental materials [21,22]. The translucency of dental ceramic is highly dependent on light scattering [23]. When the majority of light passing through a ceramic is intensely scattered and diffusely reflected, the material would appear opaque. However, if only part of the light is scattered and most of it is diffusely transmitted, the material would appear translucent [24]. The amount of light that is absorbed, reflected, and transmitted depends on the amount of crystals within the core matrix, their chemical nature, and the size of the particles in relation to the incident wavelength of light [25]. Translucency of zirconia can be affected by sintering parameters [26]. It has been previously reported that during sintering process, the heat rate did not show an influence on the mechanical properties of

zirconia [27]. On the other hand, one study showed that the holding time during the sintering process caused grain expansion [28].

The aim of this study was to investigate the effect of different final sintering temperatures with a constant heat rate and holding time on the flexural strength, translucency, and grain size of Y-TZP ceramic. The tested hypotheses were that a) the increased final sintering temperature would not decrease the flexural strength and b) relative translucency and grain size would increase with the increased sintering temperature.

2. Materials and methods

Y-TZP ceramic (Ceramill ZI, Amann Girrbach, Koblach, Austria, Lot No: FL08-04119) sintered at different final sintering parameters was tested in this study. All Y-TZP ceramic specimens were prepared in the white state using a low-speed diamond saw (Well 3241, Well Diamantdrahtsägen, Mannheim, Germany) and grounded to the final dimensions using SiC discs P220, P500, P1200, P2400 and P4000 (ScanDia, Hagen, Germany) in sequence. Specimens were sintered (LHT 02/16, Nabertherm GmbH, Lilienthal/Bremen, Germany) at a heat rate of 8°C/min to the following final sintering temperatures 1300°C (group a), 1350°C (group b), 1400°C (group c), 1450°C (group d), 1500°C (group e), 1550°C (group f), 1600°C (group g), 1650°C (group h), 1700°C (group i) with 120 min holding time.

Three-point flexural strength

Three-point flexural strength (N=198; n=22 per group) was measured according to ISO 6872: 2008 [29]. After sintering procedures, the final dimensions of all specimens were 1.2 mm x 4 mm x 25 mm.

Before the flexural strength test, the dimensions of the specimens were measured with a digital micrometer (Mitutoyo, Andover, England) to the next 0.01 mm. The specimens were tested dry at room temperature. They were then placed in the appropriate sample holder and loaded in a Universal Testing Machine (Z010, Zwick, Ulm, Germany) at a cross-head speed of 1 mm/min until failure. The flexural strength was calculated according to the following formula:

$$\sigma = 3NI/2bd^2$$

where σ : flexural strength, N: fracture load (N), l: distance between bearers (mm), b: width of the specimen (mm), d: thickness of the specimen (mm).

Relative translucency

For translucency measurements, specimens (N=90; n=10) with final dimensions of 20 mm x 20 mm x 0.7 mm in white state were produced. After sintering, the specimens had an average thickness of 0.5 ± 0.05 mm. The relative translucency was measured using a spectrophotometer (CM-2600d, Konica Minolta, Hannover, Germany) according to ISO 2471: 2008 [30]. The light source was CIE illuminant D65 brightness. The degree of luminous reflectance (Y) of the specimens between black (0) and white (100) measured the relative translucency. The relative translucency was calculated as follows:

$$\text{Relative translucency} = [(Y_B - Y_W) + 100] / 100$$

where Y_B was the value with a black background, and Y_W was the value with a white background. In all calculations, 0 was considered as totally transparent and 1 as totally opaque.

Zirconia grain size

After sintering, the surface of all specimens were polished up to 1 μm diamond suspensions (Struers, Ballerup, Denmark) and ultrasonically cleaned in isopropanol. Specimens were then gold sputtered (N=9, n=1 per group) and surface topography was evaluated under a Scanning Electron Microscopy (SEM, Carl Zeiss Supra 50 VP FESEM, Carl Zeiss, Oberkochen, Germany).

Statistical analysis

The data were analyzed using a statistical software programme (SPSS Version 15, SPSS INC, Chicago, IL, USA). Initially, the descriptive statistics were computed. One-way ANOVA was used for the analysis of flexural strength and relative translucency with respect to sintering temperature, followed by Scheffé post-hoc test ($\alpha=0.05$). Simple linear correlation between the sintering temperature and

flexural strength/relative translucency was evaluated. In all tests, p-values smaller than 5% were considered as statistically significant.

3. Results

The results of the descriptive statistics (mean, SD, 95% CI) for the flexural strength and relative translucency tests for each group are presented in Table 1.

Three-point flexural strength

The lowest mean flexural strength was observed in group i where sintering temperature was 1700°C ($p < 0.05$). Significantly higher flexural strength values ($p < 0.05$) were observed in groups sintered between 1400 and 1550°C followed by (Table 1, Fig. 1).

Relative translucency

The translucency of Y-TZP increased with the final sintering temperature (Table 1, Figs. 2). While the group a (1300°C) showed the lowest ($p < 0.05$), group i (1700°C) presented the highest translucency ($p < 0.05$).

Zirconia grain size

The grain size of Y-TZP enlarged with the increase in final sintering temperature (Figs. 3a-i). While zirconia microstructure in group a (1300°C) showed the smallest, group i (1700°C) showed the largest grain size of all tested groups.

The specimens sintered up to 1300°C and 1350°C demonstrated partially insufficient sintered areas, with a greater amount of open porosity and pore interconnectivity (Figs. 3a-b). The specimens with a final sintering temperature above 1600°C possibly produced internal stresses in the zirconia surface and thereby expanded grains were observed accompanied with hollow holes in the zirconia microstructure (Figs. 4a-f).

No correlation was found between sintering temperature and flexural strength ($R^2=0.178$) but high correlation was observed between sintering temperature and relative translucency ($R^2=0.957$) (Linear correlation).

4. Discussion

The results of this study clearly showed that the sintering temperature above 1600°C decreased the flexural strength of the tested zirconia yielding to the rejection of the first hypothesis. The increase in sintering temperature enlarged the grain size and increased the translucency. Therefore, the second hypothesis was accepted.

Many zirconia manufacturers tend to increase the final sintering temperature with the expectation of increased translucency. According to the results of this study, one must consider the fact that the flexural strength and stability of zirconia above the sintering temperature of 1550°C was decreased. It has been previously reported, that ceramics in general with lower flexural strength were more translucent than those of the ceramics with higher flexural strength [26]. In this study, the grain size of Y-TZP increased with the rise in final sintering temperature that might have resulted in grain pull out and thereby, decreased mechanical properties.

In principle, zirconia materials composed of small grains are more opaque when visible light passes through. This is due to less reflection and light absorption in spite of the greater scattering from an increase number of grains. Larger grains however, cause more light reflection and therefore light does not pass through [25]. Thus, large grains in zirconia have reduced numbers of particles per unit volume and consequently, such zirconia materials exhibit less scattering and decreased opacity [22].

The increased grain size may result in crack propagation since applied stress intensity factor may affect the transformation change [31]. When tetragonal zirconia is subjected to tension, it minimizes this perturbation by expanding its dimensions and transforming to monoclinic zirconia, as this expansion decreases the initially

applied tensile stress [1]. Based on the findings of this study, it could be stated that zirconia with larger grain size fractured earlier. Numerous studies have reported that reducing the average of grain size in Y-TZP ceramics has a beneficial effect on the stability of the tetragonal phase [32-36]. The specimens with final sintering temperature above 1600°C produced internal stress in the zirconia surface; the grains expanded until the immediate neighbouring grains were pressed out which consequently resulted in holes within the zirconia microstructure. By the rise of grain size, the internal tensile stresses in the zirconia have possibly increased. The emerging internal stresses could produce an undesired phase transformation from tetragonal to the monoclinic zirconia. The phase transformation is combined with the aging effect of zirconia and decreases the mechanical properties [1]. In summary, the stability of zirconia is more important than the aesthetic properties of the dental applications.

The clinical implication of this study may not be of that important for the veneered zirconia FDPs but for the monolithical ones having no veneering ceramic. Many studies reported that the major disadvantage of Y-TZP is its reduced stability against low temperature degradation (LTD) [31,35-38]. LTD is defined as the spontaneous tetragonal to monoclinic transformation occurring over time at low temperatures, when the transformation is not triggered by the local stress produced at the trip of the advancing crack [2]. Under hydrothermal changes, an undesired phase transformation from tetragonal phase to the weaker monoclinic zirconia can occur on the surface of the monoclinical zirconia FDPs. This is combined with an increase in the surface roughness [37] resulting possibly in high abrasion of the antagonist enamel. Moreover, through the undesired phase transformation as a result of aging, mechanical properties may decrease further [37,38].

The long-term failure probability of monolithic zirconia is of importance for dental applications. At this time, no clinical studies are available reporting this phenomenon. Nevertheless, according this study, when a compromise needs to be made for the optical and mechanical properties of such zirconia, the range of increased sintering temperatures should be taken into consideration in order not to experience possible clinical failures.

One limitation of this study was that only one zirconia brand was used. The results may not apply for other zirconia materials with different grains sizes. Future studies should verify the results of this study also under hydrothermal aging conditions.

5. Conclusions

1. Zirconia ceramic tested showed the highest flexural strength with final sintering temperatures between 1400°C and 1550°C.
2. Translucency of the tested zirconia increased with the increase in final sintering temperature.
3. Enlarged grains of the zirconia microstructure were observed at higher final sintering temperatures.
4. Sintering temperatures above 1600°C resulted in grain expansion and hollow holes in the zirconia microstructure.

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Conflict of interest

The authors declare no conflicts of interest.

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Tables

Table 1 Mean (SD) values of flexural strength and relative translucency of all tested groups.

Figures

Fig 1 Mean flexural strength of zirconia after different sintering temperatures.

Fig 2 Relative translucency of zirconia after different sintering temperatures.

Figs 3a-i Zirconia grain size after different sintering temperatures (50.000x), a) 1300°C, b) 1350°C, c) 1400°C, d) 1450°C, e) 1500°C, f) 1550°C, g) 1600°C, h) 1650°C, i) 1700°C.

Fig 4 Surface topography of sintered zirconia at a-c) 1650°C (1st row) and d-f) 1700°C (2st row).

Fig 1 Mean flexural strength of zirconia after different sintering temperatures.

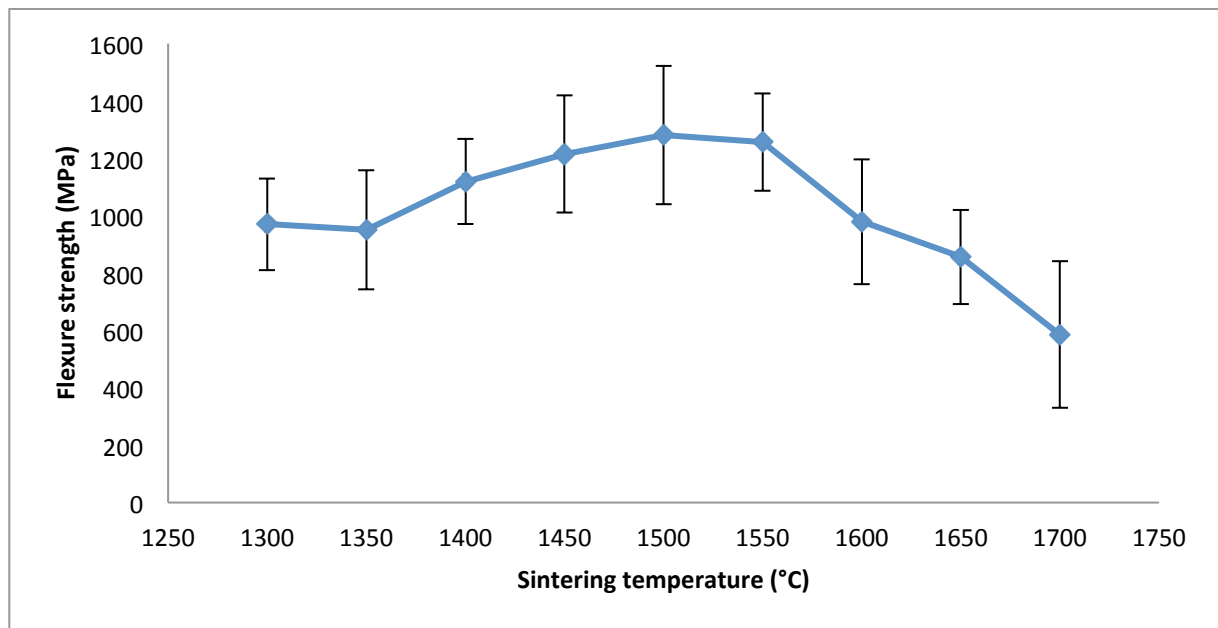


Fig 2 Relative translucency of zirconia after different sintering temperatures.

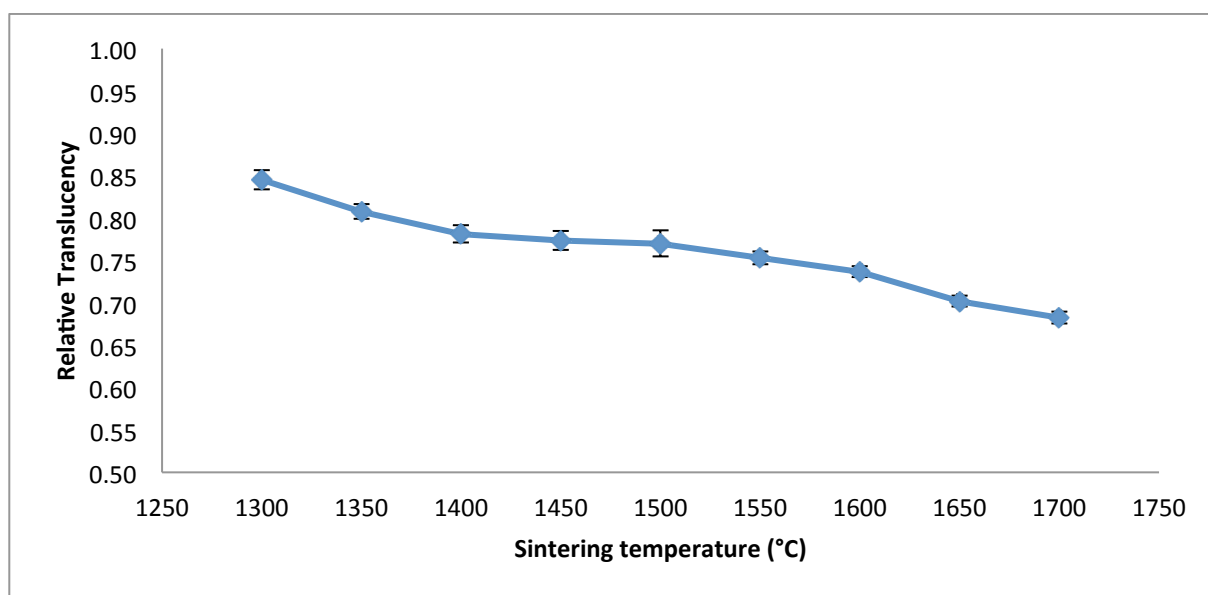


Fig 3a-i Zirconia grain size after different sintering temperatures (50.000x), a) 1300°C, b) 1350°C, c) 1400°C, d) 1450°C, e) 1500°C, f) 1550°C, g) 1600°C, h) 1650°C, i) 1700°C.

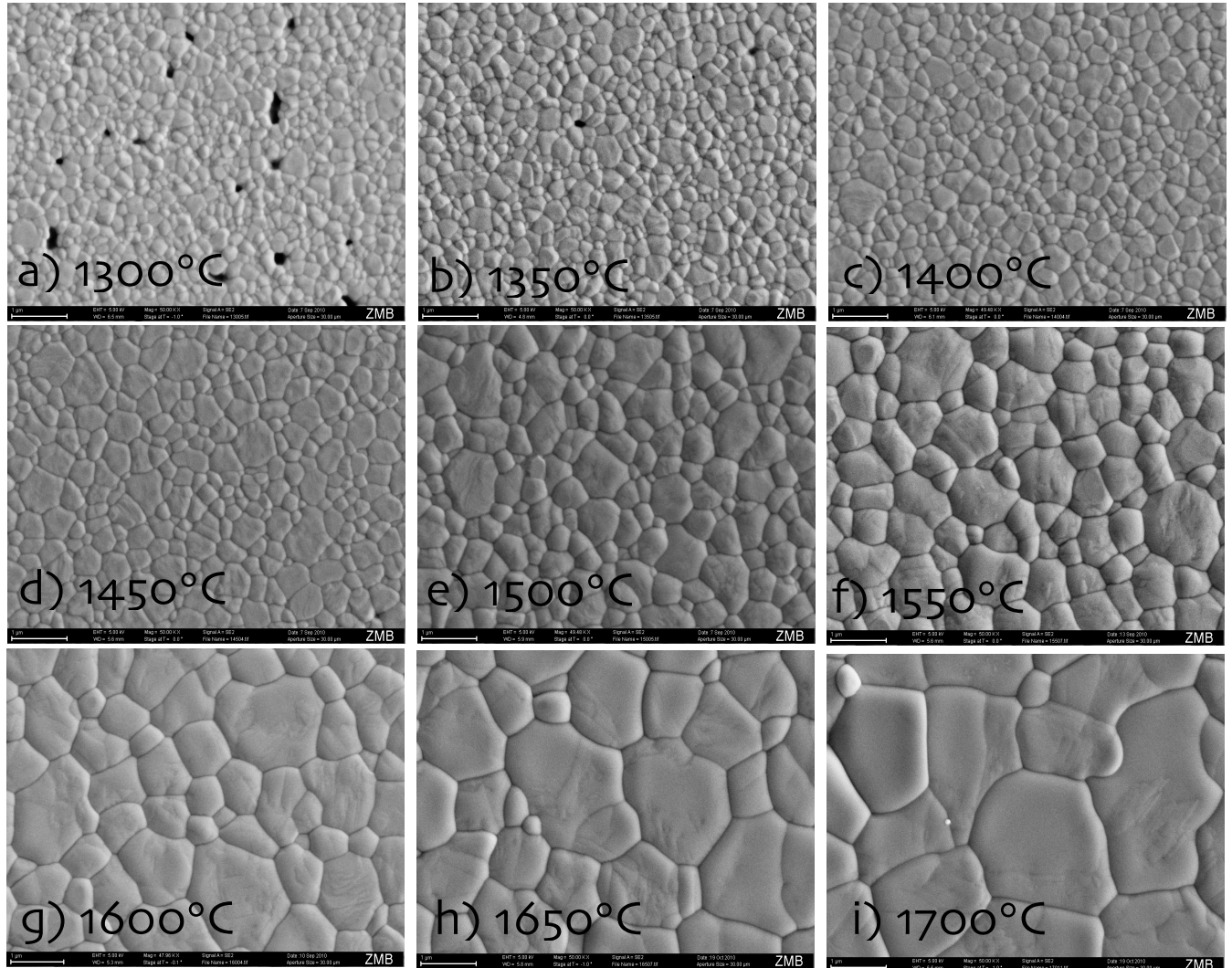


Fig 4 Surface topography of sintered zirconia at a-c) 1650°C (1st row) and d-f) 1700°C (2st row).

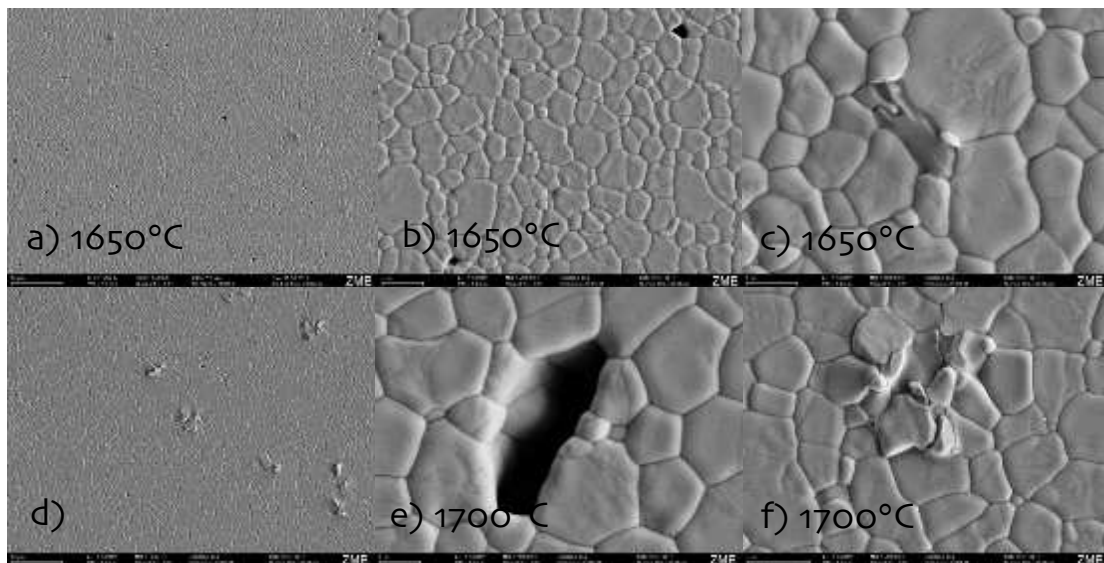


Table 1 Mean (SD) values of flexural strength and relative translucency of all tested groups.

Sintering temperature (°C)	Flexural strength (MPa)		Relative translucency	
	Mean (SD)	95% CI	Mean (SD)	95% CI
1300 (group a)	969.8 (157) ^{bc}	(898;1042)	0.85 (0.01) ^g	(0.83;0.86)
1350 (group b)	950.9 (201) ^{bc}	(861;1040)	0.81 (0.01) ^f	(0.80;0.81)
1400 (group c)	1119.3 (143) ^{cd}	(1055;1183)	0.78 (0.01) ^e	(0.77;0.79)
1450 (group d)	1214.5 (194) ^d	(1128;1301)	0.77 (0.01) ^e	(0.76;0.79)
1500 (group e)	1281.1 (230) ^d	(1179;1384)	0.77 (0.02) ^{de}	(0.76;0.79)
1550 (group f)	1256.7 (165) ^d	(1183;1331)	0.75 (0.01) ^{cd}	(0.74;0.76)
1600 (group g)	979.2 (218) ^{bc}	(882;1076)	0.74 (0.01) ^c	(0.73;0.75)
1650 (group h)	856.4 (168) ^b	(781;932)	0.70 (0.01) ^b	(0.69;0.71)
1700 (group i)	585.6 (251) ^a	(474;697)	0.68 (0.01) ^a	(0.67;0.69)

* Different super scripts represent a significant difference in each column.